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REPORT
CD NO.

50X1-HUM

COUNTRY	USSR	DATE OF INFORMATION	1948
SUBJECT	Scientific - Chemistry, toxic compounds		
HOW PUBLISHED	Bimonthly periodical	DATE DIST.	18 SEP 1950
WHERE PUBLISHED	Moscow	NO. OF PAGES	11
DATE PUBLISHED	May - Jun 1950		
LANGUAGE	Russian	SUPPLEMENT TO REPORT NO.	

THIS IS UNEVALUATED INFORMATION

SOURCE Izvestiya Akademii Nauk, SSSR, Otdeleniye Khimicheskikh Nauk, No 3, 1950.

DIPOLE MOMENTS OF ESTERS OF PHOSPHORIC, PHOSPHOROUS, AND PHOSPHONIC ACIDS

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Submitted 12 July 1948

/Physical constants of the compounds in question are of interest, because toxic derivatives of this general type have been synthesized and are being synthesized at present. Compounds of this type can also be used as intermediates in the synthesis of other substances exhibiting higher toxicity and volatility than the initial materials. The isomerization of phosphorous acid ester into alkylphosphonic acid esters merits attention in that connection.

No intention to synthesize toxic compounds on the basis of data published in this instance has been expressed by the Russian investigators in the text of the paper; the statement made above refers to potentialities only./

Introduction

Data for this article was obtained in 1940-41 in the Laboratory of Organic Chemistry, Scientific-Research Institute imeni A. M. Butlerov, but Rakov died in the Nazi invasion, and for a number of reasons, his material has been withheld from publication until now.

The authors present data which was obtained by measuring the dipole moments of 22 esters of the acids in question.

On a great number of examples the marked difference between the dipole moments of trivalent and pentavalent phosphorus derivatives could be shown. These differences make it possible to predict the valence of derivatives from the dipole moments.

Attempts to establish the presence of two tautomeric forms of dialkylphosphorous acids by measuring their dipole moments were unsuccessful.

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Previous literature has given but sparse detail on dipole moments of the derivatives of phosphorus, and particularly on those of the above esters. Smith and Lewis (1) have given some information on the dipole moments of the triphenyl ester of phosphorous acid and the triphenyl ester of thiophosphoric acid, but the accuracy of their results is doubted since they used commercial products of questionable purity in their experiments.

The authors express a particular interest in dialkylphosphorous acids, the dipole moments of which they hoped to use in establishing the presence of two tautomeric forms (representing tri- and pentavalent phosphorus):

$$HO - P(OR)_2 \quad \text{and} \quad \overset{H}{\diagdown}\!P(OR)_2 \overset{}{\underset{O}{/\!\!/}}$$

The authors expected data on the dipole moments of dialkylphosphorous acids to confirm conclusions regarding the structure of these compounds that were previously drawn from investigation of their chemical properties.

The measurement of dielectric constants of solutions was performed at 20 degrees with an Henriquez (2) apparatus made by the Kipp Firm and using a solution in carbon tetrachloride. The refraction index was determined with a Pulfrich refractometer; the density, with a Mendeleyev pycnometer which these scientists modified considerably. For the calculation of the dipole moments of solutions of a substance in carbon tetrachloride, De Vries Robles' equation was used (3):

$$\mu^2 \; 0.76378 \left(\frac{\Delta \varepsilon}{x}\right)_{x=0} - 1.1075\left(\frac{\Delta n^2{}_D}{x}\right)_{\text{average}} - 0.052593\left(\frac{dD_{12}}{dx}\right)_{x=0}$$

$$+ 0.0005446 \; M_2,$$

where x is the molar percentage of the solvent in the solution; ε, the dielectric constant; D, density, subscript 1 pertains to the solvent; subscript 2, to the dissolved polar substance; and M_2, the molecular weight of the investigated polar substance.

(With carbon tetrachloride at 20 degrees centigrade it was assumed that $n_D = 1.46048$; $D_1 = 1.5944$; $M_1 = 153.84$; $MR_D = 26.45$.)

Dipole Moments of Esters of Phosphorous Acid $P(OR)_3$

These esters were prepared according to Milobendzki's method (4) by the action of phosphorus trichloride on corresponding alcohols in an ether solution in the presence of pyridine. The esters prepared in this manner as a rule contained some diester. The esters were freed from this admixture by treatment with sodium metal (5). Milobendzki (6) established by spectroscopic investigations that the esters of phosphorous acid produced by his method contained pyridine in the ratio of 1:547.

In producing the methyl ester of phosphorous acid by Milobendzki's method, the necessary degree of purity was not obtained, so it became necessary to prepare trimethyl phosphite by the action of phosphorus trichloride on the alcoholate of methyl alcohol.

The dipole moments of triethylphosphite prepared according to Milobendzki's and Rakov's methods, within the limits of accuracy of the experiment, were proven to be similar.

Phenyl ester of phosphorous acid was prepared by the action of phosphorus trichloride on phenol; it was purified by distillation in vacuum.

Constants obtained for these esters are given in Table 6, and their dipole moments, as measured by Arbuzov and Rakov, are presented in Table 1.

As shown from the data presented, the dipole moments of trialkylphosphites change little in relation to the size of the radical, a fact which was also observed in the cases of other homologous series.

The dipole moment of triphenylphosphite found by these authors ($\mu = 1.59$ D) sharply disagrees with the value found by Smith and Lewis ($\mu = 2.08$ D), a fact which is explained by the impurity of the latter two scientists' preparation.

Dipole Moments of Esters of Phosphoric Acid

Esters of phosphoric acid also were prepared by the Milobendzki method.

The phenyl ester of phosphoric acid was prepared by the action of phosphorus oxychloride on phenol. Constants for these esters are given in Table 7, and their dipole moments are given in Table 2.

Dipole moments of phosphoric esters are considerably higher than those of phosphorous esters. The value for the dipole moment of triphenylphosphate, found by Arbuzov and Rakov ($\mu = 2.89$ D) is close to the Smith and Lewis value ($\mu = 2.82$ D).

Dipole Moments of Esters of Alkylphosphonic Acids

Esters of alkylphosphonic acids were prepared according to a method previously developed by A. Ye. Arbuzov (7) and involving isomerization of esters of phosphorous acid by the corresponding alkyl halides.

Constants of these esters are listed in Table 8. The dipole moments are shown in Table 3.

The dipole moments of alkylphosphonic esters, which are isomerous with the corresponding phosphorous esters, have high values which may be expected in the case of derivatives of pentavalent phosphorus. Dipole moments of esters of alkylphosphonic acids are on the average 0.1 D below the dipole moment of phosphoric esters and 1.0 D above those of phosphorous esters.

Dipole Moments of Dialkylphosphorous Acids

Dialkylphosphorous acids $(RO)_2POH$ were prepared by the action of phosphorus trichloride on the corresponding alcohols. Their constants are given in Table 9, and their dipole moments measured in a carbon tetrachloride solution are listed in Table 4.

As shown in Table 4, the dipole moments of dialkylphosphorous acids are close to the values for the dipole moments of phosphoric esters, which indicates that dialkylphosphorous acids, according to the values for their dipole moments, should be derivatives of pentavalent phosphorus. However, if it is assumed that dialkylphosphorous acids can be expressed by two tautomeric equations and with the equilibrium

$$HO-P(OR)_2 \rightleftarrows (O)=\underset{\displaystyle H}{\underset{|}{P}}-(OR)_2$$

displaced almost entirely to the right, it can be expected that, in different solvents, this equilibrium will more or less shift to one side. Arbuzov and Rakov determined the dipole moments of diethylphosphorous acid dissolved in carbon tetrachloride, benzene, cyclohexane, and decaline. The results are shown in Table 5, and they closely conform to each other, except for cyclohexane, the value of which is much less. However, such a decrease can be due to the effect of the solvent.

In order to illustrate the effect of cyclohexane on the dipole moment, the values for the dipole moments of triethylphosphate in two solvents are cited: in carbon tetrachloride, - 3.07 D; in cyclohexane, 2.84 D.

It is apparent that the decreased D of diethylphosphorous acid in a cyclohexane solution is due to the effect of the solvent. The data obtained on the dipole moments of dialkylphosphorous acids shows no possibility of solving the problem concerning the presence of tautomeric forms of these organic derivatives of phosphorus.

Experimental Part

The substances after preparation were stored in sealed ampoules. Before use, they were once more distilled in a vacuum. The first and last fractions of the distillate were discarded.

In computing μ according to the equation of De Vries [illegible]bles, it was necessary to determine three values: $\left(\frac{\Delta \varepsilon}{x}\right)_{x=0}$

where $\Delta \varepsilon$ is the difference of the dielectric constants of the solution and the solvent: $x = \frac{n}{N+n}$, the molar fraction; and $\frac{\Delta \varepsilon}{x}$ was extrapolated graphically to infinite dilution.

$\left(\frac{dD_{12}}{dx}\right)_{x=0}$ in all cases, except for solid phenyl ester of phosphoric acid, was determined according to the equation:

$$\left(\frac{dD_{12}}{dx}\right)_{x=0} = \frac{M_2 D_1 (D_2 - D_1)}{M_1 D_2}$$

The values $\frac{\Delta \varepsilon}{z}$, $\frac{\Delta n^2 D}{x}$, and $\frac{dD_2}{d_2}$ for individual solutions are given in tables 10-14.

For calculation of the dipole moment of diethylphosphorous acid in solvents other than CCl_4 the following facts were assumed:

For benzene at 20 degrees

$$\varepsilon = 2.2830;\ D = 0.8780;\ M = 78.05;\ n_D = 1.50093;\ \mu^2 = 0.6898;$$
$$\left(\frac{\Delta \varepsilon}{x}\right)_{x=0} - 0.6998;\ \left(\frac{\Delta n^2_{DV}}{x}\right)_{av} - 0.02381;\ \left(\frac{dD_{12}}{dx}\right)_{x=0} + 0.000258\ M_2;$$

For cyclohexane at 20 degrees

$$\mu^2 = 0.93584; \left(\frac{\Delta \varepsilon}{k}\right)_{x=0} - 0.94476; \left(\frac{\Delta n_D^2}{x}\right)_{av} - 0.0231; \left(\frac{dD_{12}}{dx}\right)_{x=0} + 0.00021\, M_2; \quad \varepsilon = 2.055;\ D = 0.7783;\ n_D^{20} = 1.42680;\ M = 84.16;$$

For decaline at 20 degrees

$$\varepsilon = 2.1300;\ D = 0.8952;\ n_D^{20} = 1.48035;\ \mu^2 = 1.289; \left(\frac{\Delta \varepsilon}{x}\right)_{x=0} - 1.25 \left(\frac{\Delta n_D^{20}}{dx}\right)_{av} - 0.03465; \left(\frac{dD_{12}}{dx}\right)_{x=0} + 0.000280\, M_2.$$

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/Tables follow./

Table 1

Esters of $P(OR)_3$	μ in D
Methyl	1.83
Ethyl	1.96
n-propyl	1.99
Iso-propyl	1.98
n-butyl	1.92
Phenyl	1.59

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Table 2

Esters of $P(O)(OR)_3$	μ in D
Methyl	3.02
Ethyl	3.07
n-propyl	3.09
Iso-propyl	2.85
n-butyl	3.05
Phenyl	2.89

Table 3

Esters of $R\text{-}P(O)(OR)_3$	μ in D
Methyl ester of methylphosphonic acid	2.86
Ethyl ester of ethylphosphonic acid	2.91
n-propyl ester of n-propylphosphonic acid	2.92
Iso-propyl ester of iso-propylphosphonic acid	2.91
n-butyl ester of n-butylphosphonic acid	2.90

Table 4

Esters of $(RO)_2$ POH	μ in D
Methyl	2.94
Ethyl	3.08
n-propyl	3.15
Iso-propyl	3.08
n-butyl	3.17

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Table 5

Diethylphosphorous Acid in Solution of	μ in D
CCl_4	3.06
Decaline	3.04
C_6H_6	3.17
Cyclohexane	2.85

Table 6. Constants of Esters of Phosphorous Acid

Substance	Bp (deg C)	Pressure (mm)	d_4^{20}	n_D^{20}	Molecular Wt
$P(OCH_3)_3$	111-112	760	1.04934	1.40895	124.08
$P(OC_2H_5)_3$	47-47.5	10	0.96867	1.41351	166.16
n.$P(OC_3H_7)_3$	85-85.5	11	0.92758	1.4244[illegible]	208.24
Iso-$P(OC_3H_7)_3$	63.0	11.5	0.89382	1.41880	208.24
n.$P(OC_4H_9)_3$	121-121.5	8	0.91334	1.43268	250.32
$P(OC_6H_5)_3$	220	11	1.18801	1.59106	310.28

Table 7. Constants of Esters of Phosphoric Acid

Substance	Bp (deg C)	Pressure (mm)	d_4^{20}	n_D^{20}	Molecular Wt
$PO(OCH_3)_3$	192-193	760	1.20482	1.39675	140.08
$PO(OC_2H_5)_3$	99.5	13	1.06771	1.40648	182.16
n.$PO(OC_3H_7)_3$	120.5-121.5	10	1.01187	1.41677	224.24
Iso-$P(OC_3H_7)_3$	105	13	0.98511	1.40663	224.24
n.$PO(OC_4H_9)_3$	127.5	3	0.97621	1.42480	266.32
$PO(PC_6H_5)_3$	m.p. 48-49	--	--	--	326.28

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Table 8. Constants of Dialkylphosphorous Acids

Substance	Bp (deg C)	Pressure (mm)	d_4^{20}	n_D^{20}	Molecular Wt
$(CH_3O_2)POH$	53.0	7	1.19441	1.40360	110.05
$(C_2H_5O)_2POH$	76.0	14	1.07560	1.40807	138.11
$(n\text{-}C_3H_7O)_2POH$	87.0	6	1.01792	1.41835	166.16
$Iso\text{-}(C_3H_7O)_2POH$	80.5	12	0.99626	1.40903	166.16
$(n\text{-}C_4H_9O)_2POH$	122.0	9	0.98499	1.42542	194.21

Table 9. Constants of the Esters of Alkylphosphonic Acids

Substance	Bp (deg C)	Pressure (mm)	d_4^{20}	n_D^{20}	Molecular Wt
$CH_3\text{-}P(O)(OCH_3)_2$	181.0	760	1.16840	1.41458	124.08
$C_2H_5\text{-}P(O)(OC_2H_5)_2$	90.0	10	1.02585	1.41647	166.16
$n\text{-}C_3H_7\text{-}P(O)(OC_3H_7)_2$	114.0	0	0.97300	1.42312	208.24
$Iso\text{-}C_3H_7\text{-}P(O)(OC_3H_7)_2$	83-83.5	10	0.96112	1.41398	208.24
$n\text{-}C_4H_9\text{-}P(O)(OC_4H_9)_2$	148.5-149.5	9	0.94968	1.43288	250.32

Table 10

Substance	x	$\Delta\varepsilon$	$\Delta\varepsilon/x$	Δn	$\frac{\Delta n^2}{x}$	$\left(\frac{dD_{12}}{dx}\right)_{x=0}$
$P(OCH_3)_3$	0	0	4.03 ± 0.01	0	-0.15	-0.668
	0.02251	0.0959	4.04	$-105\cdot10^{-3}$	-0.14	
	0.04227	0.1655	4.03	$-214\cdot10^{-3}$	-0.15	
	0.05442	0.2190	4.02	$-304\cdot10^{-3}$	-0.16	
	0.05879	0.2375	4.04	$-304\cdot10^{-3}$	-0.15	
	0.07601	0.3054	4.02	$-414\cdot10^{-3}$	-0.16	
$P(OC_2H_5)_3$	0	0	4.64 ± 0.01	0	-0.14	-1.112
	0.00833	0.0390	4.65	$-373\cdot10^{-4}$	-0.13	
	0.01271	0.0588	4.63	$-609\cdot10^{-4}$	-0.14	
	0.01376	0.0638	4.64	$-707\cdot10^{-4}$	-0.15	
	0.02116	0.0983	4.65	$-101\cdot10^{-3}$	-0.14	
$n.P(OC_3H_7)_3$	0	0	4.72 ± 0.02	0	-0.13	-1.553
	0.00492	0.0232	4.74	$-236\cdot10^{-4}$	-0.14	
	0.01103	0.0526	4.73	$-456\cdot10^{-4}$	-0.12	
	0.02077	0.0975	4.70	$-434\cdot10^{-3}$	-0.13	
	0.03032	0.1426	4.70	$-545\cdot10^{-3}$	-0.13	
	0.04175	0.1962	4.70	$-830\cdot10^{-2}$	-0.15	

Table 10 (Contd)

Substance	x	$\Delta\varepsilon$	$\Delta\varepsilon/x$	Δn	$\frac{\Delta n^2}{x}$	$\left(\frac{dD_{12}}{dx}\right)_{x=0}$
Iso-$P(OC_3H_7)_3$	0	0	4.68 ± 0.01	0	-0.15	-1.670
	0.01196	0.0532	4.45	$-66\cdot10^{-3}$	-0.16	
	0.02100	0.0900	4.29	$-115\cdot10^{-3}$	-0.16	
	0.03012	0.1241	4.12	$-165\cdot10^{-3}$	-0.16	
n.$P(OC_4H_9)_3$	0	0	4.32 ± 0.02	0	-0.12	-1.935
	0.00792	0.0396	4.34	$-35\cdot10^{-4}$	-0.13	
	0.03715	0.1588	4.30	$-326\cdot10^{-4}$	-0.12	
	0.04278	0.1847	4.31	$-176\cdot10^{-3}$	-0.12	
$P(OC_6H_5)_3$	0	0	4.44 ± 0.01	0	+1.00	+1.00
	0.00619	0.0273	4.43	--	--	--
	0.01111	0.0495	4.45	--	--	
	0.01396	0.0621	4.45	$+482\cdot10^{-3}$	+1.27	
	0.01784	0.0795	4.45	$+702\cdot10^{-3}$	+1.23	
	0.01802	0.0803	4.45	$+750\cdot10^{-3}$	+1.21	
	0.02197	0.0978	4.45	$+888\cdot10^{-3}$	+1.188	
	0.03446	0.1526	4.43	$+134\cdot10^{-2}$	+1.148	

Table 11

Substance	x	$\Delta\varepsilon$	$\Delta\varepsilon/x$	Δn	$\frac{\Delta n^2}{x}$	$\left(\frac{dD_{12}}{dx}\right)_{x=0}$
$PO(OCH_3)_3$	0	0	11.54 ± 0.02	0	-0.20	-0.469
	0.00882	0.1035	11.73	$-55\cdot10^{-4}$	-0.18	
	0.01764	0.2110	11.97	$-135\cdot10^{-3}$	-0.18	
	0.02675	0.3385	11.22	$-174\cdot10^{-3}$	-0.20	
	0.03471	0.4295	12.32	$-254\cdot10^{-3}$	-0.21	
	0.03680	0.4564	12.40	$-260\cdot10^{-3}$	-0.20	
$PO(OC_2H_5)_3$	0	0	11.89 ± 0.02	0	-0.19	-0.931
	0.00482	0.0574	11.90	$-314\cdot10^{-4}$	-0.19	
	0.00985	0.1181	11.98	$-607\cdot10^{-4}$	-0.18	
	0.01036	0.1237	11.94	$-709\cdot10^{-4}$	-0.20	
	0.01459	0.1748	11.98	$-106\cdot10^{-3}$	-0.21	
	0.02264	0.2723	12.03	$-147\cdot10^{-3}$	-0.19	
n.$PO(OC_3H_7)_3$	0	0	12.02 ± 0.02	0	-0.18	-1.338
	0.00914	0.1101	12.04	$-60\cdot10^{-3}$	-0.19	
	0.01372	0.1647	12.00	$-75\cdot10^{-3}$	-0.18	
	0.02110	0.2540	12.04	$-146\cdot10^{-3}$	-0.20	
	0.02324	0.2789	12.00	$-143\cdot10^{-3}$	-0.18	
Iso-$PO(OC_3H_7)_3$	0	0	10.08 ± 0.01	0	-0.19	-1.437
	0.00523	0.0522	10.09	$-34\cdot10^{-3}$	-0.19	
	0.01012	0.1020	10.08	$-73\cdot10^{-3}$	-0.21	
	0.01532	0.1543	10.08	$-94\cdot10^{-3}$	-0.18	
	0.01810	0.1823	10.07	$-118\cdot10^{-3}$	-0.19	
	0.01871	0.1884	10.07	$-122\cdot10^{-3}$	-0.19	
n.$PO(OC_4H_9)_3$	0	0	11.91 ± 0.01	0	-0.17	-1.748
	0.00701	0.0834	11.90	$-55\cdot10^{-4}$	-0.15	
	0.01266	0.1509	11.92	$-105\cdot10^{-3}$	-0.17	
	0.02383	0.2837	11.91	$-135\cdot10^{-3}$	-0.17	
	0.03011	0.3585	11.90	$-194\cdot10^{-3}$	-0.19	
$PO(OC_6H_5)_3$	0	0	12.16 ± 0.01	0	+1.10	-2.013
	0.00304	0.0371	12.19	$+304\cdot10^{-3}$	+1.38	
	0.00560	0.0682	12.19	$+403\cdot10^{-3}$	+1.12	
	0.00887	0.1082	12.21	$+473\cdot10^{-3}$	+1.00	

Table 12

Substance	x	$\Delta\varepsilon$	$\Delta\varepsilon/x$	Δn	$\frac{\Delta n^2}{x}$	$\left(\frac{dD_{12}}{dx}\right)_{x=0}$
$(CH_3O)_2P\text{-}OH$	0	0	10.98 ± 0.03	0	-0.16	-0.382
	0.01800	0.2074	11.52	$-100\cdot10^{-3}$	-0.16	
	0.02937	0.3482	11.86	$-154\cdot10^{-3}$	-0.16	
	0.03129	0.3718	11.91	$-160\cdot10^{-3}$	-0.16	
	0.03283	0.3919	11.94	$-158\cdot10^{-3}$	-0.15	
$(C_2H_5O)_2P\text{-}OH$	0	0	12.04 ± 0.01	0	-0.15	-0.690
	0.01489	0.1801	12.14	$-82\cdot10^{-3}$	-0.16	
	0.01743	0.2122	12.17	$-90\cdot10^{-3}$	-0.15	
	0.02517	0.3077	12.23	$-129\cdot10^{-3}$	-0.15	
	0.02789	0.3417	12.25	-1[illegible]3·10^{-3}	-0.16	
	0.02954	0.3624	12.27	$-152\cdot10^{-3}$	-0.15	
$(n\text{-}C_3H_7O)\ P\text{-}OH$	0	0	12.62 ± 0.02	0	-0.14	-0.975
	0.00602	0.0762	12.66	$-29\cdot10^{-4}$	-0.14	
	0.01229	0.1554	12.68	$-55\cdot10^{-4}$	-0.13	
	0.01823	0.2324	12.75	$-97\cdot10^{-4}$	-0.15	
	0.02462	0.3148	12.78	$-118\cdot10^{-3}$	-0.14	
	0.02998	0.3839	12.81	$-144\cdot10^{-3}$	-0.14	
Iso-$(C_3H_7O)_2P\text{-}OH$	0	0	12.03 ± 0.02	0	-0.16	-1.034
	0.00794	0.0956	14.04	$-35\cdot10^{-4}$	-0.13	
	0.01366	0.1643	12.02	$-68\cdot10^{-4}$	-0.16	
	0.02173	0.2612	12.02	$-120\cdot10^{-3}$	-0.16	
	0.02759	0.3326	12.05	$-179\cdot10^{-3}$	-0.19	
$(n\text{-}C_4H_9O_2)\text{-}P\text{-}OH$	0	0	12.73 ± 0.02	0	-0.13	-1.245
	0.00535	0.0681	12.73	$-24\cdot10^{-3}$	-0.13	
	0.01089	0.1385	12.72	$-45\cdot10^{-3}$	-0.13	
	0.01603	0.2042	12.75	$-71\cdot10^{-3}$	-0.13	
	0.02093	0.2659	12.71	$-95\cdot10^{-3}$	-0.13	
	0.02594	0.3297*	12.71	$-115\cdot10^{-3}$	-0.13	

*[The Russian text gives the erroneous figure 03.297]

Table 13

Substance	x	Δx	$\Delta\varepsilon/x$	Δn	$\frac{\Delta n^2}{x}$	$\left(\frac{dD_{12}}{dx}\right)_{x=0}$
$CH_3PO(OCH_3)_2$	0	0	10.35 ± 0.01	0	-0.14	-0.469
	0.01297	0.1362	10.50	$-45\cdot10^{-4}$	-0.10	
	0.02301	0.2436	10.59	$-105\cdot10^{-3}$	-0.13	
	0.02713	0.2892	10.66	$-125\cdot10^{-3}$	-0.14	
	0.03563	0.3816	10.73	$-164\cdot10^{-3}$	-0.14	
	0.04062	0.4410	10.79	$-204\cdot10^{-3}$	-0.15	
$C_3H_5PO(OC_2H_5)_2$	0	0	10.74 ' 0.01	0	-0.13	-0.954
	0.01138	0.1227	10.76	$-51\cdot10^{-2}$	-0.13	
	0.01204	0.1298	10.78	$-54\cdot10^{-3}$	-0.13	
	0.01850	0.2001	10.79	$-87\cdot10^{-3}$	-0.14	
	0.03697	0.4011	10.85	$-165\cdot10^{-3}$	-0.13	
	0.04445	0.4847	10.90	$-198\cdot10^{-3}$	-0.13	

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Table 13 (Contd)

Substance	x	Δx	Δε/x	Δn	$\frac{\Delta n^2}{x}$	$\left(\frac{dD_{12}}{dx}\right)_{x=0}$
$C_3H_7PO(OC_3H_7)_2$	0	0	10.77 ± 0.02	0	-0.12	-1.378
	0.00975	0.1051	10.78	$-40 \cdot 10^{-3}$	-0.12	
	0.01820	0.1958	10.76	$-75 \cdot 10^{-3}$	-0.12	
	0.01905	0.2054	10.79	$-78 \cdot 10^{-3}$	-0.12	
	0.03391	0.3644	10.75	$-139 \cdot 10^{-3}$	-0.12	
	0.04955	0.5322	10.75	$-203 \cdot 10^{-3}$	-0.12	
Iso-$C_3H_7PO(OC_3H_7)_2$	0	0	10.64 ± 0.01	0	-0.15	-1.422
	0.00378	0.0402	10.63	$-19 \cdot 10^{-3}$	-0.15	
	0.00737	0.0785	10.65	$-38 \cdot 10^{-3}$	-0.15	
	0.01365	0.1452	10.53	$-65 \cdot 10^{-3}$	*	
	0.02110	0.2245	10.64	$-108 \cdot 10^{-3}$	-0.15	
	0.02610	0.2780	10.63	$-134 \cdot 10^{-3}$	-0.15	
n-$C_4H_9PO(OC_4H_9)_2$	0	0	10.57 ± 0.01	0	-0.08	-1.761
	0.01925	0.2038	10.58	$-53 \cdot 10^{-3}$	-0.08	
	0.02513	0.2653	10.56	$-69 \cdot 10^{-3}$	-0.08	
	0.02920	0.3086	10.57	$-85 \cdot 10^{-3}$	-0.08	

*[Illegible; probably 0.14]

Table 14

Substance	x	Δε	Δε/x	Δn	$\frac{\Delta n^2}{x}$	$\left(\frac{dD_{12}}{dx}\right)_{x=0}$
$(C_2H_5O)_2POH$						
In benzene	0	0	14.30 0.01	0	-0.27	+0.286
	0.00747	0.1093	14.64	$-187 \cdot 10^{-3}$	-0.27	
	0.01462	0.2187	17.96	$-187 \cdot 10^{-3}$	-0.25	
	0.01499	0.2249	14.99	$-188 \cdot 10^{-3}$	-0.27	
	0.02222	0.3409	15.34	$-196 \cdot 10^{-3}$	-0.27	
	0.02237	0.3427	15.33	$-201 \cdot 10^{-3}$	-0.27	
In cyclohexane	0	0	8.53 ± 0.01	0	-0.08	+0.353
	0.00857	0.0828	8.60	$-24 \cdot 10^{-3}$	-0.08	
	0.00963	0.0739	8.62	$-27 \cdot 10^{-3}$	-0.08	
	0.01699	0.1475	8.68	$-48 \cdot 10^{-3}$	-0.08	
	0.02638	0.2308	8.75	$-74 \cdot 10^{-3}$	-0.08	
	0.03442	0.3045	8.85	$-96 \cdot 10^{-3}$	-0.08	
	0.04297	0.3832	8.92	$-120 \cdot 10^{-3}$	-0.08	
In decaline	0	0	6.00 ± 0.01	0	-1.13	+0.144
	0.01362	0.0814	5.98	--	--	
	0.02516	0.1561	6.20	$-112 \cdot 10^{-2}$	-1.29	
	0.03659	0.2291	6.26	$-118 \cdot 10^{-2}$	-0.95	
	0.03956	0.2496	6.31	$-119 \cdot 10^{-2}$	-0.88	
	0.04935	0.3150	6.38	$-132 \cdot 10^{-2}$	-0.68	
	0.06001	0.3879	6.46	$-137 \cdot 10^{-2}$	-0.67	
$PO(OC_2H_5)_3$						
In cyclohexane	0	0	8.50 ± 0.01	0	-0.07	+0.391
	0.00967	0.0818	8.[illegible]	$-24 \cdot 10^{-3}$	-0.07	
	0.01972	0.1685	8.54	$-48 \cdot 10^{-3}$	-0.07	
	0.02801	0.2431	8.68	$-69 \cdot 10^{-3}$	-0.07	
	0.03774	0.3300	8.75	$-93 \cdot 10^{-3}$	-0.07	
	0.04097	0.3591	8.77	$-100 \cdot 10^{-3}$	-0.07	

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